

1363 783

- (21) Application No. 49488/72 (22) Filed 26 Oct. 1972  
 (61) Patent of Addition to No. 1 313 085 dated 7 Sept. 1971  
 (31) Convention Application No. P 21 55 696.7  
 (32) Filed 9 Nov. 1971 in  
 (33) Germany (DT)  
 (44) Complete Specification published 14 Aug. 1974  
 (51) International Classification C07G 7/026  
 (52) Index at acceptance C3H 2



(54) PROCESS FOR THE PRODUCTION OF A WHEY  
 PROTEIN CONCENTRATE

(71) I, JOSEF ANTON MEGGLE, a German citizen, trading as Molkerei J. A. Meggle, Milchindustrie of 8094 Reitmehring, Germany, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention is concerned with a process for the production of a whey protein concentrate.

In main Patent Specification No. 1,313,085, there is described and claimed a process for the production of a whey protein concentrate by ultrafiltration, wherein whey is adjusted to a pH value below the isoelectric point of the whey protein, thereafter subjected to ultrafiltration and heated to ensure microbial destruction before and/or after the ultrafiltration.

Ultrafiltration is a modern process which permits the separation of colloidal particles from solvents. The separation takes place by means of ultrafiltration membranes of small pore diameter which permit a separation of substances with molecular weights above about 20,000.

Since ultrafiltration is an especially gentle process, it can be used for the production of protein concentrates from whey. However, it has been shown that sour whey, i.e. whey which normally has a pH between 4.2 and 4.8, can only be ultrafiltered so slowly that the value of the process is questionable from an economic point of view. The ultrafiltration of sweet whey, i.e. whey with a pH between 5.5 and 6.5, is a known process but it suffers from the disadvantage that the cleaning of the membranes from protein and mineral material residues causes difficulties and, in addition, the growth of micro-organisms in this pH range can proceed unhindered. The period of time of the known ultrafiltration process until the desired degree of protein concentration

is obtained, is comparatively great, which necessarily results in a contamination of the product obtained due to the high content of micro-organisms.

The fact that sour whey, i.e. whey with a pH at about the isoelectric point of the whey protein, is particularly difficult to ultrafilter, also makes it difficult to ultrafilter mixtures of sweet and sour whey. As is known, both kinds of whey are very frequently obtained in dairies and it is in the interest of dairies to be able to work up both kinds of whey without selection. Furthermore, in the course of storage or transport, sweet whey often becomes sour which, when it comes to working it up, places the whey in an unfavourable condition for ultrafiltration.

It is an object of the present invention to remove these difficulties and to provide a process for the ultrafiltration of whey which permits such ultrafiltration rates to be achieved that an optimum and economically acceptable use of the process is possible and, at the same time, to reduce the danger of a contamination by micro-organisms.

According to the present invention, this problem is solved by an improvement or modification of the process of my main Patent No. 1,313,085. Thus, according to the present invention, there is provided a process for the production of a whey protein concentrate, wherein whey is adjusted to a pH below the isoelectric point of the whey protein, preferably to a pH between 2.7 and 3.3, and then subjected to ultrafiltration at ambient temperature or with heating.

I have found that, in the given pH range, a substantial acceleration of the ultrafiltration takes place, in comparison with a pH at about the isoelectric point. Furthermore, the cleaning of the membrane parts at these pH values no longer presents a problem, in contradistinction to processes carried out at a neutral pH or at a pH at about the isoelectric point.

At the same time, the rate of growth of micro-organisms is so reduced that the above-mentioned disadvantage of bacterial contamination in the case of known processes is overcome.

5 Furthermore, it is surprising that, in contradistinction to the process carried out at a neutral pH or at a pH at about the isoelectric point, an increase of the ultrafiltration temperature also results in increased throughput rates. Utilisation of this discovery makes it possible further to reduce the danger of infection during the ultrafiltration. The ultrafiltration according to the present invention is, therefore, preferably carried out with heating at a temperature between 40 and 65°C.

10 For the selection of the pH value at which the process according to the present invention is to be carried out, it is important that it is lower than the lowest isoelectric point of the protein components of the whey. This means that the ultrafiltration must be carried out at least below the isoelectric point of the main amount of the protein components of the whey, i.e. below the precipitation maximum for the whey protein under the usual conditions of heat denaturing. Small amounts of protein with a still lower isoelectric point do not noticeably disturb the process. Best results are, however, achieved between pH 2.7 and pH 3.3. The adjustment of the pH value to the range used according to the process of the present invention takes place in known manner. Preferably, the pH value is adjusted by cation exchange because this results in a further improvement of the ultrafiltration capacity. Examples of preferred methods for the adjustment of the pH value by cation removal include the use of ion exchanger and electrodialysis.

20 As already mentioned above, the process according to the present invention results in a substantial reduction of the time necessary for the ultrafiltration of whey. In the case of sour whey, it is now possible to carry out the ultrafiltration while heating, with simultaneous increasing of the throughput. It also provides, for the first time, the possibility of working up by means of ultrafiltration to a protein concentrate, under reproducible conditions and with reproducible results, whey of differing origin and pretreatment, i.e. concentrated, partially desaccharised, partially desalinated or partially deproteinised whey, because, by means of this process, the whey protein is converted into a uniform, quasi-dissolved state, which is the same as the state in which it is originally present.

25 As also already mentioned above, a further advantage of the process of the present invention is that the cleaning of the ultrafiltration membrane is simplified. I have found that the membrane, when working under the pH conditions used according to the process of the present invention, can be adequately cleaned by a simple acidic cleaning at a pH of 2 or below. A cleaning with the use of proteolytic

enzymes, such as is generally recommended for the technique of ultrafiltration, is thus normally not necessary at all. When, on the other hand, whey is ultrafiltered at its inherent pH and at an elevated temperature, then the cleaning of the membrane causes considerable difficulties and can practically not be carried out successfully without the use of proteolytic enzymes.

30 The process of the present invention can be used not only for pure whey but also for mixtures of whey with other casein-containing milk derivatives, for example buttermilk, without losing the advantages of the new process. The only prerequisite is that the pH of the mixture must also be maintained below the isoelectric point so that the casein is present in a dissolved or dispersed state.

35 The products obtained by the process according to the present invention can be widely used in the production of foodstuffs. For example, they can be used for the preparation of cakes with curd fillings, products with any desired high proportion of whey protein being a valuable supplement for natural curds. Especially when additional milk protein has been introduced, as explained above in detail, there is obtained a product which is outstandingly suitable for the production of special protein-rich foodstuffs. The smooth consistency and creaminess of the product can be utilised for the preparation of dips, emulsions, such as salad dressing and mayonnaise-like products, as well as similar delicatessen products. The products of the process of the present invention can also be used as dietetic foodstuffs.

40 The following Examples are given for the purpose of illustrating the present invention:—

#### Example 1.

Sour whey was ultrafiltered at pH 3 and a temperature of 16°C. A dry mass throughput of 13.7 g./m<sup>2</sup>/minute was hereby measured.

45 The process was repeated at a temperature of 50°C. The dry mass throughput was then 16g/m<sup>2</sup>/minute.

For comparison, the same sour whey was ultrafiltered at its natural pH (pH 4.5). At 16°C., a dry mass throughput of 7.7 g./m<sup>2</sup>/minute was obtained, and at 50°C., an ultrafiltration of 8.8g/m<sup>2</sup>/minute.

50 Thus, when using the process according to the present invention, the throughput rate was almost doubled.

#### Example 2.

Sweet whey of pH 6.5 was ultrafiltered at 16°C. A throughput of, on average, 11 g./m<sup>2</sup>/minute was obtained.

55 Under otherwise the same conditions but at a temperature of 50°C there were obtained throughputs of 8.3 and 11.7 g/m<sup>2</sup>/minute.

The above experiment was repeated but,

according to the process of the present invention, the sweet whey was first adjusted to pH 3. At 16°C., an average throughput of about 13 g./m<sup>2</sup>/minute was obtained. Under otherwise the same conditions but at a temperature of 50°C., throughput rate of up to 24g/m<sup>2</sup>/minute were obtained.

WHAT I CLAIM IS:—

1. A process for the production of a whey protein concentrate, wherein whey is adjusted to a pH below the isoelectric point of the whey protein and then subjected to ultrafiltration at ambient temperature or with heating.

2. A process according to claim 1, wherein the ultrafiltration is carried out at a pH of 2.7 to 3.3.

3. A process according to claim 1 or 2, wherein the pH is adjusted to below the isoelectric point by cation exchange.

4. A process according to any of the pre-

ceding claims, wherein the ultrafiltration is carried out while heating to a temperature between 40 and 65°C.

5. A process according to any of the preceding claims, wherein the whey is used in admixture with a casein-containing milk derivative.

6. A process according to claim 1 for the production of a whey concentrate, substantially as hereinbefore described and exemplified.

7. Whey protein concentrates, whenever produced by the process according to any of claims 1 to 6.

VENNER, SHIPLEY & CO.,  
Chartered Patent Agents,  
Rugby Chambers,  
2, Rugby Street,  
London, WC1N 3QU.  
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1974.  
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

**THIS PAGE BLANK (USPTO)**